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A VOLUMETRIC METHOD FOR TITRIMETRIC ANALYSIS OF HYDROGEN PEROXIDE

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This report documents an oxidation-reduction scheme for the volumetric analysis of hydrogen peroxide in the approximate range of 30 percent, but it is not limited to this range.

The purpose of the proposed method is to provide a titrimetric, non-instrumental method for the analysis of hydrogen peroxide.

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FOREWORD

This report describes a titrimetric method (using ferrous-dichromate oxidation reduction) of analysis for hydrogen peroxide. The concept is theoretically sound and should allow for the analysis of this material over a wide range of concentrations.

The purpose of this method is to provide a means of quantitative analysis by laboratories that do not have access to instrumentation capable of analyzing hydrogen peroxide.

Funding for this work was provided by Naval Sea Systems Command, Code 644.

Approved by:

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TABLES

INTRODUCTION

The literature for three possible methods of determining hydrogen peroxide was reviewed. The military specification (Reference 1) MIL-H-22868 (Wep) uses potassium permanganate as the volumetric reagent. While the use of potassium permanganate is perfectly acceptable in most cases, it cannot be used if chloride anion is present in solution since chlorine gas will be liberated and give erroneous results. It is also unstable and must be restandardized at frequent intervals. This method is described in greater detail elsewhere (References 2, 3).

The direct titration of hydrogen peroxide with tetravalent cerium (References 4, 5) was the second method reviewed. Oxidimetry titration with ceric ion has certain advantages over potassium permanganate, particularly in respect to its greater stability and its lesser tendency to oxidize chloride ions. In titration of reducing substances that are colorless in solution, the yellow color of the excess ceric ions serves as a fairly satisfactory indicator. Sulfuric acid solutions of cerium are stable over long periods of time, unlike the less stable nitric and hydrochloric acid solutions.

The third method reviewed is work done at Sanda, Inc. (Reference 6) for the analysis of manganese dioxides. The original Sanda method involves the reaction of manganese dioxides with excess ferrous ion. The excess ferrous ion is then back titrated with dichromate using the thermometric titration technique as a means of detecting the equivalence point.

A modification, devised in this laboratory, of the third method was selected as the best approach since the potassium dichromate reagent used is more stable than the reagents used in the other methods. The modification involves the reaction of hydrogen peroxide with excess ferrous ion. The excess ferrous ion is then back titrated with dichromate using diphenylamine sulfonate as indicator. The chemistry (References 7 through 10) of the reactions involved in the modified method have been throughly investigated and are well documented.

CAUTIONARY NOTES: READ BEFORE PROCEEDING

1. References to a particular company's product line neither constitutes an endorsement nor a recommendation for use. The examples are used only as a method of illustrating the purity of reagents and the quality of apparatus needed for the method.

2. Unless otherwise indicated, the word water means distilled water (e.g., glass still).

3. Chemicals and apparatus listed in this method may be substituted with chemicals of a higher purity or apparatus of a better quality. However, chemicals of a lesser purity and apparatus of a lower quality may not be substituted for those specified in this method.

4. Unless otherwise stated, all chemicals used in this method are certified ACS reagent grade chemicals.

5. This method was developed for the analysis of 30 percent hydrogen peroxide. However, it can be adapted to analyze other concentrations by proper adjustment of the normality of the volumetric reagents, or changing the sample weight taken for analysis.

6. Hydrogen peroxide is quite often decomposed due to contamination of glassware and equipment. Therefore, all glassware and equipment used for analysis of hydrogen peroxide shall be properly cleaned prior to use.

7. All chemicals have some degree of hazard. This method requires the same due care in handling as any chemical procedure where injury could result from improper handling techniques.

REAGENTS

1. Ferrous ammonium sulfate hexahydrate. For example, Fisher number I 77-500.

2. Ferrous sulfate heptahydrate. For example, Fisher number I 146-500.

3. Potassium dichromate. For example, Fisher number P 188-500.

4. Diphenylamine sulfonic acid sodium salt oxidation-reduction indicator. For example, Fisher number D141-5.

5. Sulfuric acid, concentrated (95-98 percent). For example, Fisher number A 300-212.

6. O-Phosphoric acid, 85 percent. For example, Fisher number A 242-500.

7. 5 N sulfuric acid (Reference 11): Slowly pour 75 mL of concentrated sulfuric acid (95-98 percent) into approximately 200 mL of water. Allow to cool and dilute to 500 mL.

8. Ferrous ammonium sulfate, 0.50 normal (Reference 6): Slowly pour 100 mL of concentrated sulfuric acid into approximately 500 mL of water in a 1000 mL beaker. While the solution is still hot add 196.08 g of ferrous ammonium sulfate hexahydrate. (If ferrous ammonium sulfate hexahydrate is not

available 139.01 g of ferrous sulfate heptahydrate may be substituted.) Stir, with a stirring rod, until the salt is dissolved. Quantitatively transfer the solution to a 1000 mL volumetric flask, wash the beaker three times and add the washings to the volumetric flask. Allow the solution to cool to room temperature and dilute to 1000 mL.

9. Potassium Dichromate, 0.20 Normal (Reference 11): Dry approximately 10 g of chemically pure ACS grade potassium dichromate, at 150 degrees celsius, to a constant weight. Cool in a desiccator and weigh approximately 9.8 g of potassium dichromate to the nearest 0.1 mg. Quantitatively transfer the salt to a 1000 mL volumetric flask and add about 500 mL of water. Swirl until the salt is completely dissolved. Dilute to 1000 mL and mix well. The normality is calculated in the following manner. Normality = weight of dichromate divided by 49.032.

10. Diphenylamine sulfonate indicator solution: Dissolve 0.14 g of diphenylamine sulfonic acid sodium salt in 100 mL of water.

COMPARISON OF FERROUS SOLUTION TO DICHROMATE SOLUTION

Immediately prior to analysis of the hydrogen peroxide the ferrous solution should be titrated with the dichromate solution as follows. Accurately measure 4, 5 and 6 mL of 0.5 N ferrous solution into separate 500 mL titration flasks and dilute to about 150 mL. Add 10 mL of 5 N sulfuric acid, 5 mL of 85 percent phosphoric acid and 20 drops of diphenylamine sulfonate indicator solution to each flask. Dilute to about 200 mL and titrate, while stirring, with the 0.2 N dichromate solution. Near the equivalence point the green color will begin to deepen. Continue to titrate until the deepening green color turns abruptly to an intense violet color. This violet color (almost the color of grape juice) is the equivalence point and should remain violet for a minimum of 30 seconds. Record the volumes of dichromate required to titrate the three given volumes of ferrous solution. These values will be used to calculate an average dichromate-ferrous ratio which is needed to determine the percent hydrogen peroxide.

PROCEDURE

Using a 5 mL disposable beaker weigh accurately between 1.1500 to 1.3500 g of the hydrogen peroxide sample. Quantitatively transfer the weighed sample to a 500 mL titration flask and dilute to 150 mL. Add 50 mL of 0.5 N ferrous solution, 10 mL of 5 N sulfuric acid, 5 mL of 85 percent phosphoric acid and 20 drops of the diphenylamine sulfonate indicator. Titrate, while stirring, with 0.2 normal dichromate solution.

Just before the equivalence point the green color will begin to deepen. Carefully continue the titration until there is an abrupt change from green to violet. This violet color is the equivalence point and should remain for a minimum of 30 seconds. Record the weight of peroxide taken for analysis and the volumes of ferrous and dichromate solutions used. Average the results from a minimum of three titrations to determine the concentration of hydrogen peroxide.

CALCULATIONS

HP = hydrogen peroxide.
(NF) = normality of ferrous solution.
(ND) = normality of dichromate solution.
(MF1) = milliliters of ferrous solution used to determine dichromate-ferrous ratio.
(MF2) = milliliters of ferrous solution used for reaction with HP sample.
(MD1) = milliliters of dichromate solution used to determine dichromate-ferrous ratio.
(MD2) = milliliters of dichromate solution used to titrate excess ferrous ion after reaction with the HP.
(S) = sample weight of 30 percent HP taken for analysis.
(WH) = milliequivalent weight of HP (0.017007g).
(MF2)(NF) = total milliequivalents of ferrous solution used for reaction with HP sample.
(MD2)(ND) = milliequivalents of dichromate solution used to titrate the excess ferrous solution.

I. [(MF2)(NF)] - [(MD2)(ND)] = milliequivalents of ferrous solution reacted wit
HP = milliequivalents of HP taken for analysis.

II. % HP = (100)(weight of HP)/(S).

III. Weight of HP = [(MF2)(NF) - (MD2)(ND)](WH)
substitution of III into II

IV. % HP = 100[(MF2)(NF) - (MD2)(ND)](WH)/(S)
At the equivalence point (MF1)(NF) = (MD1)(ND)
(NF) = (MD1)(ND)/(MF1)
Let (MD1)/(MF1) = (RC) = comparison ratio

V. Then (NF) = (ND)(RC) substitution of V into IV
% HP = 100[(MF2)(ND)(RC) - (MD2)(ND)](WH)/(S)
% HP = 100(ND)[(MF2)(RC) - (MD2)] (WH)/(S)
Since (WH) = 0.017007
Then 100 (WH) = 1.7007

VI. % HP = 1.7007(ND)[(MF2)(RC) - (MD2)]/(S)

CRITERIA FOR REJECTION OF DATA POINTS

Absolutely no data should be discarded unless statistical criteria clearly show them to be in error. When the analyst is aware that deviations from prescribed experimental procedure have occurred, the data should be discarded. Otherwise, data suspected of being in error can be tested by calculating its T value. If the calculated T value is greater than that listed in Table 1, the value may be rejected.

$$Tn = (Xn - Xb)/s$$

where Tn = T for the observation being tested
Xn = individual value being tested
Xb = arithmetic average of all values in the group being tested
s = standard deviation of the group being tested

TABLE 1. CRITICAL VALUES FOR T

Number of Observations	Significance Level		
n	5%	2.5	1%
3	1.15	1.15	1.15
4	1.46	1.48	1.49
5	1.67	1.71	1.75
6	1.82	1.89	1.94
7	1.94	2.02	2.10
8	2.03	2.13	2.22

Calculation of standard deviation

$$s = [\sum_{i=1}^{N} (Xi - Xb)^2/n-1]^{1/2}$$

n = number of values
Xi = each value in the level containing Xn
Percent coefficient of variation = $s/Xb \times 100$

RESULTS

PRELIMINARY DATA

A dichromate solution was prepared according to paragraph 9 under Reagents Section, and the calculated normality was = 0.2003.

Dichromate-Ferrous comparison ratio

(MF1)	(MD1)	(RC)	Drops of indicator
5.0	12.40	2.48	10
10.0	24.90	2.49	10
15.0	37.35	2.49	10

dichromate-ferrous ratio average = 2.4867
standard deviation = 0.0058
coefficient of variation = 0.2322

Sample analysis

Mallinckrodt # 5239 with stabilizer: (MF2) = 50

Sample #	(S)	(MD2)	% HP	Drops of indicator	Tn
1	0.97	35.05	31.36	10 smokey grey	1.00
2	1.54	sample too large			----
3	0.87	40.80	32.71	10 smokey grey	1.64
4	1.36	6.60	29.49	20 violet	0.12
5	1.23	24.74	27.58	30 deep violet	0.78
6	1.31	14.25	28.63	20 violet	0.29
7	1.31	16.26	28.10	10 smokey grey	0.54
8	1.03	43.20	26.83	20 smokey grey	1.14

average % HP = 29.24
standard deviation = 2.11
coefficient of variation = 7.22

Sample # 6 calculation

$$\% \text{ HP} = 1.7007(\text{ND})[(\text{MF2})(\text{RC}) - (\text{MD2})]/(\text{S})$$

$$\% \text{ HP} = 1.7007(0.2003)[(50)(2.4867) - (14.25)]/1.31 = 28.63$$

COMMENTS: Samples used to gather the preliminary data were weighed to the nearest 10 mg on a top loading balance. This was done because it was more rapid and the data were used only to determine if the method would work. Ability to see the equivalent point accurately is affected by the amount of indicator and the volume of dichromate used. During analysis it was found that the optimum amount of indicator to use was 20 drops (about 0.6 mL). Also the titration volume of dichromate should be controlled between 5 and 25 mL. The volume of dichromate used is controlled by the amount of hydrogen peroxide taken for analysis.

FINAL DATA

Day 1

Dichromate-Ferrous Comparison Ratio

(MF1)	(MD1)	(RC)
4.0	10.0	2.50
5.0	12.6	2.52
6.0	15.0	2.50

average RC = 2.5067
standard deviation = 0.0114
coefficient of variation = 0.4607

Sample Analysis

Mallinckrodt # 5239 with stabilizer: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP	Tn
1	1.3102	9.17	30.20	1.11
2	1.2394	20.97	28.69	0.99
3	1.3756	5.0	29.80	0.55
4	1.3156	13.65	28.92	0.67

average % HP = 29.40
standard deviation = 0.72
coefficient of variation = 2.44

Mallinckrodt # 5240 no stabilizer: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP	Tn
1	1.3205	23.5	26.27	0.52
2	1.3329	21.6	26.51	1.13
3	1.4150	16.3	26.25	0.65

average % HP = 26.35
standard deviation = 0.15
coefficient of variation = 0.55

Fisher # H355 - Lot 714337: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP
1	1.3290	15.4	28.18
2	1.2777	19.70	28.16
3	1.3680 ran out of ferrous solution		

average % HP = 28.17

Day 2

Dichromate-Ferrous Comparison Ratio

(MF1)	(MD1)	(RC)
4.0	10.00	2.5000
5.0	12.40	2.4800
6.0	15.10	2.5167

average RC = 2.4989
standard deviation = 0.0184
coefficient of variation = 0.7347

Sample Analysis

Mallinckrodt # 5239 with stabilizer: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP	Tn
1	1.3156	10.60	29.61	1.16
2	1.2579	15.9	29.53	0.79
3	1.2769	14.20	29.55	0.30

average % HP = 29.56
standard deviation = 0.04
coefficient of variation = 0.14

Mallinckrodt # 5240 no stabilizer: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP	Tn
1	1.4660	12.13	26.22	0.88
2	1.4416	11.47	26.82	0.19
3	1.4212	8.5	27.91	1.08

average % HP = 26.99
standard deviation = 0.86
coefficient of variation = 3.19

Fisher # H325 Lot 714337: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP	Tn
1	1.3478	9.68	29.13	1.02
2	1.3169	13.82	28.75	0.94
3	1.3326	11.83	28.92	0.07

average % HP = 28.93
standard deviation = 0.19
coefficient of variation = 0.67

Fisher # H325 Lot 720017: (MF2) = 50 mL

Sample #	(S)	(MD2)	%HP	Tn
1	1.3047	14.40	28.86	0.93
2	1.3211	11.55	29.24	1.07
3	1.3330	11.45	29.04	0.16

average % HP = 29.04
standard deviation = 0.19
coefficient of variation = 0.66

APPARATUS

1. 500 mL titration flasks, at least six. For example, Fisher number 10-091C or Kimble number 26650.

2. 100, 500, and 1000 mL class A volumetric flasks, at least two each. For example, Corning number 5580 or Fisher numbers 10-202B, 10-202E and 10-202F.

3. 150, 250, 600 and 1000 mL beakers, at least one each. For example, Corning number 1000 or Fisher numbers 02-540J, 02-540K, 02-540M and 02-540P.

4. 5 mL disposable polystyrene beakers, one for each weighing. For example, Fisher number 02-544-30.

5. Electric oven capable of heating to at least 200 degrees Celsius for extended periods of time. For example, Fisher #13-245-126G.

6. Analytical balance capable of weighing to at least 0.1 mg. For example, Sartorius number 1702 MP8 or Fisher number 01-636-20.

7. Top loading balance capable of weighing to at least 10 mg. For example, Sartorius number 1507 MP8 or Fisher number 01-601-42.

8. Weighing bottles, tall cylindrical form, 16 mL, 25 X 50 mm. For example, Fisher number 03-415C.

9. Flint glass pasture pipets. For example, Fisher number 13-678-6B.

10. Rubber bulbs for pasture pipets. For example, Fisher number 14-065B.

11. Bulb type safety pipet filler. For example, Fisher number 13-681-50.

12 Volumetric pipets, class A, at least one of each.

4 mL	Fisher number 13-650E
5 mL	13-650F
6 mL	13-650G
10 mL	13-650L
15 mL	13-650M
50 mL	13-650S

13. 50 mL buret with PTFE stopcock. For example, Kimble number 17094F or Fisher number 03-765.

14. Double buret clamp. For example, Fisher number 05-779.

15. Porcelain buret support. For example, Fisher number 14688.

16. Stirring bars 38 X 7.9 mm. For example, Fisher number 14-511-64.

17. Stir-light magnetic stirrer. For example, Fisher number 14-511-100. This is helpful but not necessary. If the work area is well lighted a stirrer such as the Fisher number 14-494-120MR may be used. If no stirrer is available, the solution can be carefully swirled by hand.

18. Small diameter vacuum desiccator. For example, Fisher number 08-632 or Corning number 3118 with a 140 mm plate for desiccator. Fisher number 08-641A.

19. Desiccant for desiccator. Drierite, indicating 8 mesh. For example, Fisher number 07-578-3A.

20. Graduated cylinders. 10, 50, and 100 mL, at least two each. For example, Kimble #20024 or Fisher numbers 08-549-5B, 5D and 5E.

DISCUSSION

The preliminary data were gathered to determine any variables which might affect the results. It was not expected that these data would give a good measure of the precision of the method. However, the T test showed that the results had a good precision which indicates that the method was properly designed and has good reliability. These results were supported by the final data.

Twenty drops (about 0.6 mL) of indicator gave the best results when the volume of dichromate used was maintained between 5 to 25 mL. The dark green color developed from larger amounts of dichromate tended to mask the violet color of the indicator and the equivalence point was hard to determine. When smaller amounts of indicator were used, the violet color tended not to develop and only a washed out smokey grey or smokey blue color change occurred thus making it very difficult to determine the equivalence point. The amount of O-phosphoric acid prescribed should be rigidly adhered to since less acid than this will prevent the violet color of the indicator from developing rapidly.

The reaction between hydrogen peroxide and ferrous ion was very rapid and no waiting time was necessary before titrating with potassium dichromate. Further supporting data, using the proper amount of indicator showed excellent precision and only one data point could be rejected on the basis of the T test recommended by ASTM Standard Practice D 2777-77 (Reference 12). A definite advantage of this method is that potassium dichromate can easily be obtained as a high purity salt and standardization of the volumetric reagent is not necessary when dried and carefully weighed.

CONCLUSION

With proper care, application of the method described in this technical report provides excellent precision (< 3 percent) and thus reliability for the analysis of hydrogen peroxide.

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